3M Chemicals

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8EHQ - 0898 - 14239 S

3M

August 11, 1998

Document Processing Center (7407)
(Attn: Section 8(e) Coordinator)
Office of Toxic Substances
US EPA
401 M Street, SW
Washington, DC 20460

TSCA 8(E) SUBSTANTIAL RISK NOTICE ON:
3,3,3-trifluoro-2-(trifluoromethyl)-propanoyl fluoride [382-22-9]

Dear Sir:

Pursuant to Section 8(e) of the Toxic Substances Control Act I wish to inform you of test results generated on 3,3,3-trifluoro-2-(trifluoromethyl)-propanoyl fluoride [382-22-9].

Inhalation testing of the above chemical in mice indicated a 10 min LC_{50} of less than 300 ppm (v/v) (see attachment). The purity of the test sample is indicated on the attached analytical report.

Please contact me at 651-733-0439 for further information.

Sincerely,

Charles Reich
Group Vice President
3M Chemical Markets Group
220-13W-38

Attachments:

Report on Acute Inhalation Toxicity Screen of 3,3,3-trifluoro-2-(trifluoromethyl)-propanoyl fluoride
^{1}H and ^{19}F NMR spectra of sample

Medicine, Health Physics
Industrial Hygiene, Toxicology
3M Medical Department

3M Center, Building 220-2E-02
St. Paul, MN 55144-1000
612 733 1110

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T-7009.1

Acute Inhalation Toxicity Screen for: 2-Hydrido-Perfluoroisobutryl Fluoride

3M Strategic Toxicology Laboratory
Building 270
St. Paul, Minnesota

SANITIZED

Purpose:

This test material was evaluated at the request of SCD/SMD management, as this material is present in Toxicity studies performed on similar chemicals suggest this test material may be strikingly toxic by the inhalation route. Therefore, testing needed to be performed to confirm this anticipated hazard.

Experimental Procedures:

Test Material:

Other names 3,3,3-trifluoro-2-(trifluoromethyl)-propanoyl fluoride
CAS#: 382-22-9

Material was supplied by
Chemical characterization of this material was performed using 1H-NMR and 19F-NMR by the 3M Specialty Adhesives & Chemicals Analytical Laboratory/SMD. Results of analysis are attached.

Species Selection:

Male mice will be used as the test organism.

Dose Levels:

Target concentrations of 30000 ppm, 3000 ppm, and 300 ppm were achieved by adding 390 mL, 39 mL and 3.9 mL, respectively, of the vaporized material into a 13 liter inhalation chamber.

Results:

30,000 ppm Exposure:

All three mice were dead within 5 minutes from the start of exposure. Labored breathing was observed prior to death. No necropsy was performed.

3,000 ppm Exposure:

All three mice survived the 10 minute exposure but exhibited signs of pulmonary distress (labored breathing) shortly after initiation of exposure. All three animals died within 30 minutes of the end of exposure. No necropsy was performed.

300 ppm Exposure:

Both mice survived the 10 minute exposure. Labored breathing was observed only near the end of exposure. The animals died during the night, probably 14 to16 hours after the end of the exposure. No necropsy was performed.

Discussion:

It was observed during sampling of the vapors that there was a change in pressure in the test material cylinder. This raised the possibility that the toxicity test result may be attributable to an extremely toxic impurity in the headspace. Consequently, the test material was returned to for further analysis. Subsequent analysis revealed no significant levels of other highly toxic materials in the headspace. Therefore, it is concluded that the toxicity observed in this study is the result of exposure to the title compound.

Conclusion:

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The mouse inhalation toxicity for this material is to be reported as:

10 min LC50 < 300 ppm (v/v)

Paul H. Lieder

Principle Investigator:
Paul H. Lieder, Ph.D., D.A.B.T.

July 30, 1998
DATE

3M SPECIALTY ADHESIVES & CHEMICALS ANALYTICAL LABORATORY / SMD

Request # **55258**

To:

From: Tom Kestner - (3-5633) - SA&C Analytical Lab - 236-2B-11

Subject: **Chemical Characterization of $(CF_3)_2$-CH-CO-F by ^{1}H-NMR and ^{19}F-NMR Spectroscopy**

Date: June 7, 1998

SAMPLE DESCRIPTION:

•114574-72 for toxicity testing; Nominal product = $(CF_3)_2CH$-CO-F (received in a stainless steel cylinder)

INTRODUCTION:

This sample was subjected to ^{19}F and ^{1}H-NMR spectral analyses for the determination of the purity of the nominal product. Special emphasis was placed on identifying and quantifying as many impurity components as possible. Sheila Kromer also performed a GC/MS analysis, and her results were reported to you previously.

EXPERIMENTAL:

A small aliquot (≈1.0 ml) of the neat sample liquid was transferred directly out of the stainless steel cylinder into a glass NMR tube. The sample liquid was then spiked with small amounts of $CFCl_3$ for use as the ^{19}F chemical shift zero reference, and 1,4-bis(trifluoromethyl)benzene (p-HFX) for use as a cross integration standard. Use of the p-HFX as the cross integration standard facilitated the cross correlation of the various fluorine and proton signal intensities for evaluation of the overall sample composition. 400 MHz ^{1}H-NMR and 376 MHz ^{19}F-NMR spectra were acquired using a Varian UNITYplus 400 FT-NMR spectrometer.

RESULTS:

The combined ^{19}F and ^{1}H-NMR spectral data were used to verify that the major component was the nominal product, and to determine the identities (at least tentatively) of many of minor impurity. A ^{1}H/^{19}F-NMR cross integration technique was then used to estimate the purity of the nominal product, and the concentrations of the various identified impurities. The overall qualitative and quantitative compositional data which were derived from the ^{1}H/^{19}F-NMR cross integration analyses are summarized in **TABLE-1**. The relative wt.% concentrations shown in TABLE-1 should be very close to their respective absolute wt.% values. Trace amounts of other unidentified components are also detected in the NMR spectra, but additional work would be required in an effort to identify or quantify these unassigned components. Copies of the NMR spectra are not attached with this report, but they can be provided for you if needed. If you have any questions about these results, please let me know.

Tom Kestner

File Reference: MC55258.DOC/51

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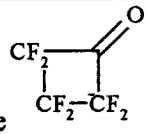
TABLE-1
Sample: 114574-72
Overall Quantitative Compositional Results by $^{1}H/^{19}F$-NMR Cross Integration

Component Structures	$^{1}H/^{19}F$-NMR Relative Wt.% Concentrations
$(CF_3)_2CH\text{-}CO\text{-}F$	99.58%
$CH_3\text{-}F$	0.20%
Possible $(CF_3)_2CH\text{-}CO_2H$	0.058%
Possible $CF_3(CF_2)_5CF_3$	0.037%
Probable $CH_3\text{-}O\text{-}CF_2\text{-}CF(CF_3)_2$	0.031%
Probable $CH_3\text{-}O\text{-}CF_2\text{-}CF_2\text{-}CF_2\text{-}CF_3$	0.026%
Possible $CF_3\text{-}CF{=}CF\text{-}CO\text{-}F$	0.022%
Possible 	0.020%
SiF_4	0.0076%
Probable $(CH_3)_2\text{-}SiF_2$	0.0063%
Probable $(CH_3)_3\text{-}Si\text{-}F$	0.0021%
$(CF_3)_2\text{-}C{=}C{=}O$	None Detected
$F_2C{=}C(CF_3)\text{-}CO\text{-}F$ (methacryloyl fluoride)	None Detected

* Trace amounts of other unassigned components are also present.

Proton NMR Spectral Assignments

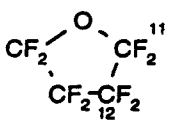
Request No: 55258 | Date: 7-6-98 | Analyst: Tom Kestner

Sample ID: 114574-72 | ^{19}F-NMR Spectra: F55258.401 | ^{1}H-NMR Spectra: H55258.401 | Solvent: Neat | Shift Ref.: $CFCl_3$

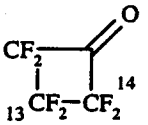
Structural Assignments

$(CF_3)_2$-CH-CO-F CH_3-F $(CF_3)_2$-CH-CF_2-O-CH_3
1 A 2 B 3 4 C 5 D

CF_3CF_2-CO_2H $CF_3CF_2CF_2$-CO_2H
6 7 8 9 10



Possible



$CF_3CF_2CF_2CF_2$-O-CH_3 $(CF_3)_2$-CF-CF_2-O-CH_3
15 16 17 18 E 19 20 21 E

$(CH_3)_2$-Si-F_2 $(CH_3)_3$-Si-F SiF_4
F 22 G 23 24

Possible $CF_3CF_2CF_2CF_2CF_2CF_2CF_3$
25 26 27 28

Possible $(CF_3)_2$-CH-CO_2H Possible CF_3-CF=CF-CO-F
29 30 31 32 33

CF_3-C_6H_4-CF_3 (internal standard)
34 H

^{19}F-NMR Chemical Shifts (-) ppm upfield from $CFCl_3$

1	-64.7 "t"
2	+50.3
3	-269.4 q
4	-64.0
5	-73.2
6	-83.7 s
7	-122.6 s
8	-81.4 t
9	-127.3 s
10	-120.1 q
11	-86.1 s
12	-132.3 s
13	-127.7 s
14	-113.6 s
15	-81.3 t
16	-126.4
17	-125.9
18	-88.7
19	-73.7
20	-187.2
21	-82.1 m
22	-132.3 septet ???
23	??
24	-164.8 s
25	-81.17 m
26	-126.1
27	-122.2
28	-121.5
29	-63.45 d
30	-69.40 d/d
31	?
32	?
33	+52.8 m
34	-64.0 s

^{1}H-NMR Chemical Shifts

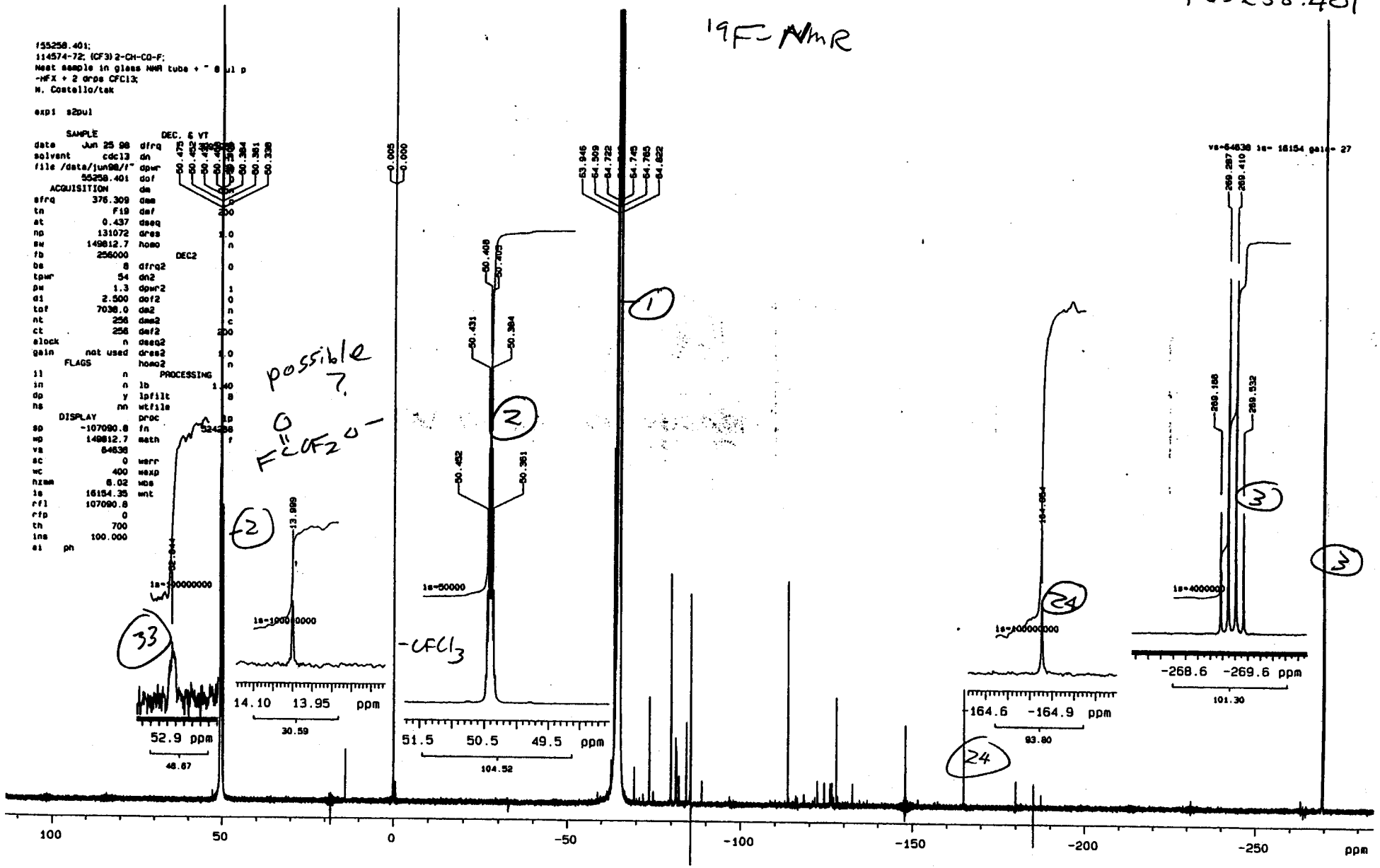
A	4.10 m
B	4.20 d
C	3.50 m
D	3.60 s
E	3.65 s
F	0.23 t
G	0.15 d
H	7.70 by definition

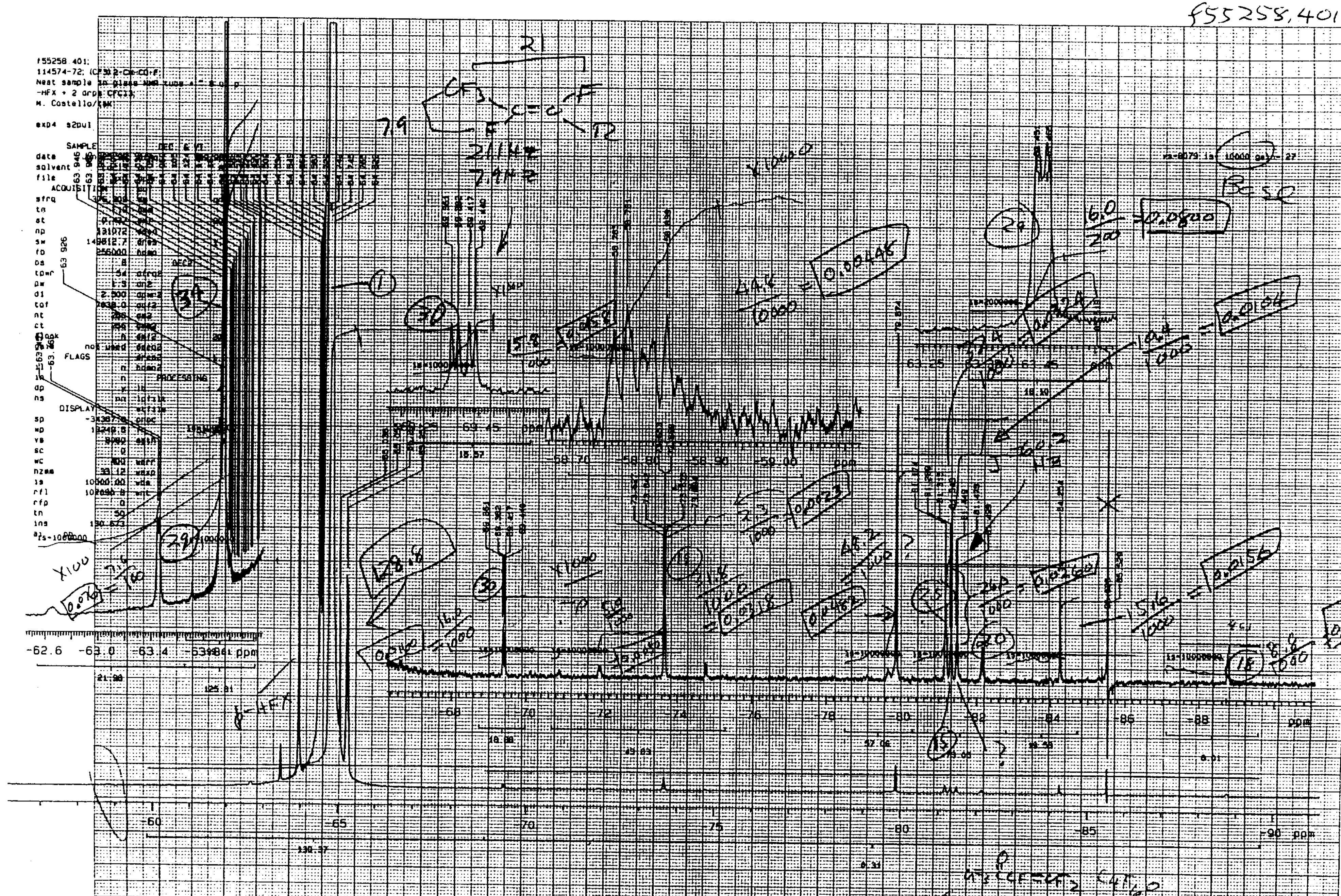
Coupling Constants (Hz)

$^{2}J_{3,B} = 48.3$;

f55258.401

^{19}F NMR





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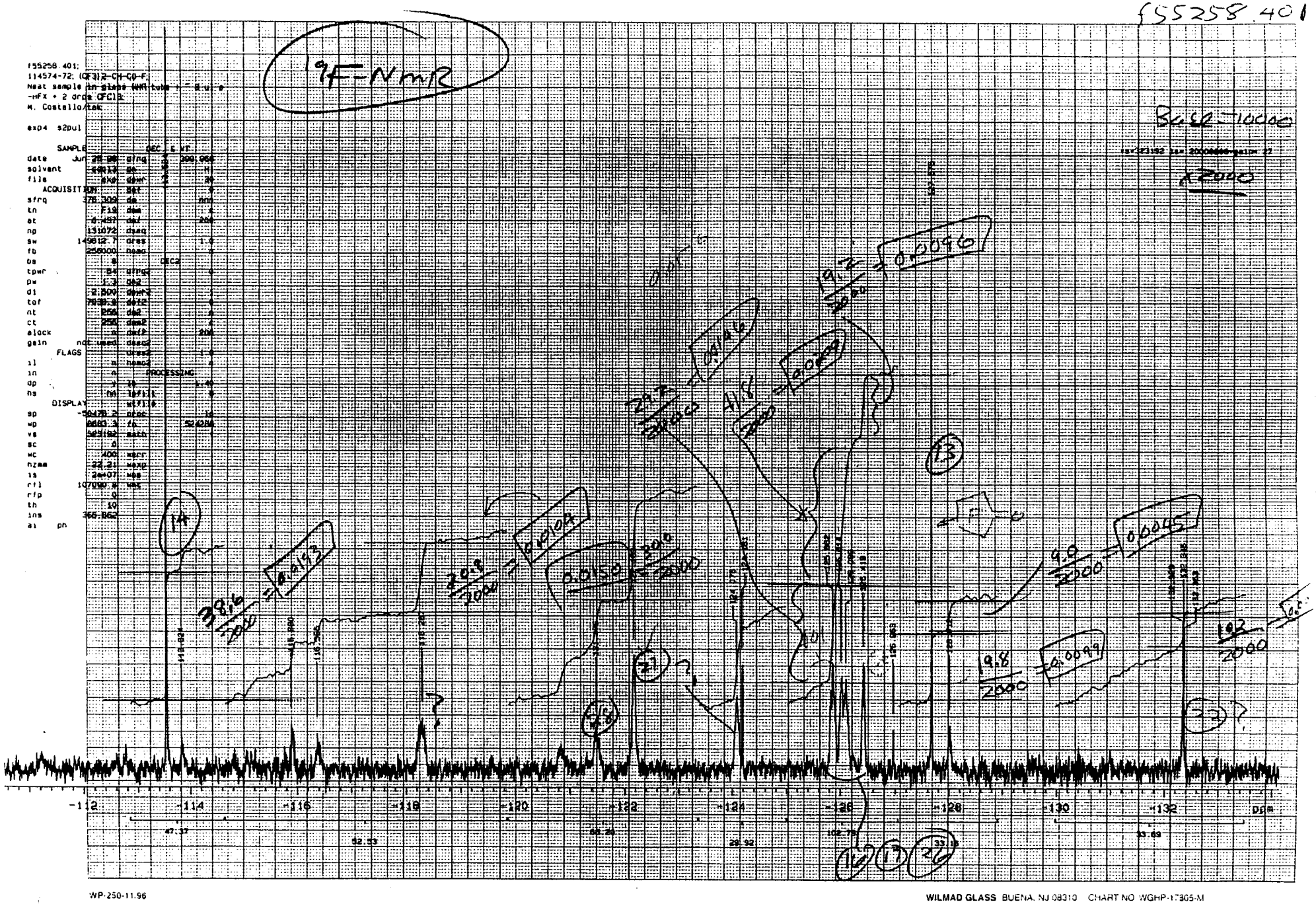

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